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(54) **Powders and products of tantalum, niobium and their alloys**

(57) Disclosed is a powder of tantalum, niobium, or an alloy thereof, having an oxygen content less than about 300 ppm and a powder metallurgy product formed therefrom. The production of the powder or body is effected without exposure to a temperature greater than about $0.7 T_H$, where T_H is the homologous temperature of the metal. The process involves blending the metal powder with a metal (e.g. magnesium) having a higher affinity for oxygen than the powder in a vacuum chamber and heating to a temperature less than about $0.7 T_H$. The metal containing the oxygen is then removed by evaporation at low pressure and subsequently by selective leaching or dissolution of the powder.

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POWDERS AND PRODUCTS OF TANTALUM,
NIOBIUM AND THEIR ALLOYS

The present invention relates to powders and products of tantalum, niobium, and their alloys having low oxygen contents, and processes for producing same.

Tantalum, and niobium are generally extracted from their ores in the form of powders. For example, tantalum is generally produced by reducing potassium fluorotantalate (K_2TAF_7) by chemical reaction with

sodium. This reduction reaction generally produces a salt-encapsulated metal powder which is crushed and washed, with water and acid, to produce tantalum powder.

Tantalum and niobium metals, and their alloys, are then consolidated to form products. The method chosen for consolidation depends upon whether the resulting consolidated product will be pure metal or an alloy, what form or shape is required, and how the material is to be used. Tantalum, niobium and their alloys are generally used to form wrought products, such as bars, plates, sheets, wire, tubes and rods; preforms, for subsequent thermo-mechanical processing; and near net shapes, for use, in a variety of applications, after machining and finishing.

Tantalum, niobium and their alloys generally have a high affinity for oxygen. Thus the oxygen content of products of niobium, tantalum or their alloys tends to increase during their formation. The oxygen content of the product affects its mechanical properties and fabricability. Generally, as the oxygen content of the product increases, the product's ductility decreases and the product's strength increases. For many applications utilizing products of tantalum, niobium or their alloys, a high oxygen content is unsuitable. Therefore, to produce tantalum, niobium or alloy products suitable for these applications, a low oxygen content must be obtained.

There are several methods which may be utilized to produce formed products of tantalum, niobium or their alloys. For example, in one method the metal is first melted by electron beam or vacuum arc melting, in a vacuum, and then thermo-mechanically processed to form the product. The melting temperature is also referred to as the homologous temperature (T_H) in degrees Kelvin. T_H for tantalum is 3273 degrees K and T_H for niobium is 2745 degrees K. The melting in a vacuum reduces the oxygen content of the metal.

In a second method the metal, in powder form, is first cold isostatically pressed into a tantalum, niobium or alloy preform, such as a bar or rod, and then the preform is resistance sintered at a temperature greater than $0.7 \frac{T}{H}$ to produce a formed product of tantalum,

niobium or their alloys. Generally, for resistance sintering, the ends of the preform are clamped between water cooled copper terminals in a high vacuum chamber and then the preform is heated, to a temperature above $0.7 \frac{T}{H}$, by passing an electrical current through the

preform. The resistance sintering simultaneously densifies and lowers the oxygen content of the preform.

However, there are many disadvantages in utilizing resistance sintering to densify and remove oxygen.

First, resistance sintering may only be utilized to produce products of certain limited shapes, generally bars or rods. For resistance sintering, the cross-section of the preform must be uniform along the path of electrical current in order to prevent localized overheating and hot-shortening. Additionally, the cross section must be small enough so that the oxygen reduction in the centre of the preform occurs before the disappearance of the interconnected porosity. For effective oxygen removal, preforms greater

than about 3.81cm (1.5 inches) in their shortest dimension are not resistance-sintered. Still further the preform must be small enough to prevent sagging associated with creep and hot pressing during unsupported resistance sintering. Thus, the preforms generally do not weigh greater than about 40lbs.

5 A third method for producing formed products of tantalum, niobium, or their alloys, is the rotating electrode process. In this process a bar or rod of the metal is heated to a temperature above T_H . The molten

metal is converted into powder by centrifugal force. The low oxygen content of the starting rod is maintained in the powder, however the powder particles are relatively spherical and generally coarser than the initial chemically produced powders. These relatively spherical powder particles are not desirable for unidirectional mechanical pressing. Further, the coarseness of the powder particles makes the powder undesirable for cold-isostatic pressing into formed tantalum, niobium or alloy products.

20 We have discovered new powders of tantalum, niobium or alloys of tantalum or niobium having an oxygen content of less than 300 ppm. We have also discovered a method

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for producing these powders wherein tantalum, niobium or alloy powders are heated in the presence of an oxygen-active metal, such as magnesium, at a temperature less than about $0.7 T_H$.

5 We have further discovered formed powder metal products having oxygen contents less than about 300 ppm formed from tantalum, niobium, and their alloys. We have still further discovered a new process for producing formed powder metal products of tantalum, niobium and
10 their alloys, having oxygen contents below 300 ppm, which is carried out without exposing the metal to a temperature greater than $0.7 T_H$.

According to the present invention, tantalum, niobium, or alloys of tantalum or niobium, powders, having
15 oxygen contents less than 300 ppm are produced by heating a tantalum, niobium, or alloy powder to a temperature lower than $0.7 T_H$ in the presence of an oxygen active

metal for a period of time sufficient to lower the oxygen content of the starting powder to less than 300 ppm.
20 Furthermore, according to the present invention, formed products of tantalum, niobium and their alloys, having oxygen contents less than 300 ppm are produced by consolidating a tantalum, niobium, or alloy powder,

having an oxygen content of less than 300 ppm, without exposing the metal to a temperature greater than about 0.7 T_H. If the starting metal powder has an oxygen

content greater than 300 ppm, then the powder must first
5 be deoxidized to a level of less than 300 ppm, such as by the technique described above. For tantalum powder, 0.7 T_H equals 2018 degrees C (2291 degrees K) and for niobium powder, 0.7 T_H equals 1650 degrees C (1923 degrees K).

10 An advantage of the powder of the present invention is that it comprises relatively non-spherical particles well suited for unidirectional mechanical pressing.

A further advantage of the powder of the present invention is that it comprises relatively small particles
15 well suited for cold-isostatic-pressing.

An advantage of the formed products of tantalum, niobium or their alloys, of the present invention, having oxygen contents less than 300 ppm, is that the products can be of any shape, cross-section or size..

20 An advantage of the process for producing formed products of the present invention is that the process allows for the production of tantalum, niobium, or alloy products having an oxygen content less than 300 ppm, of any shape, cross-section or size.

The tantalum, niobium, or alloy of tantalum or niobium powders, having an oxygen content below 300 ppm (parts per million), of the present invention, are produced by the following procedure. A tantalum, niobium or alloy powder, such as one produced by a sodium reduction process, is placed into a vacuum chamber which also contains a metal having a higher affinity for oxygen than the powder. Preferably, the starting powder has an oxygen content less than about 1000 ppm. One such metal, more oxygen active than the powder, is magnesium. The chamber is then heated, to a temperature not greater than about $0.7 T_H$, to produce a powder of tantalum, niobium or alloy of tantalum or niobium having an oxygen content less than 300 ppm. The heating is continued for a time sufficient to allow oxygen to diffuse out of the metal powder and yield a metal powder having less than 300 ppm oxygen. The magnesium, containing the oxygen, is then removed from the metal powder by evaporation, and subsequently by selective chemical leaching or dissolution of the powder.

The alloys of tantalum or niobium of the present invention include alloys of tantalum and/or niobium and an oxide which has a higher free energy of formation than tantalum oxide, such as for example yttrium oxide,

thorium oxide, or aluminium oxide. The oxide is blended into the tantalum and/or niobium powder having an oxygen content of less than 300 ppm. The alloys of the present invention also include alloys of tantalum and/or niobium and an alloying element with a low oxygen content blended into the tantalum or niobium powder, provided that the oxygen content of the blend is less than 300 ppm. The alloys of the present invention further include alloys of tantalum and/or niobium and an alloying element wherein the alloying element and the tantalum and/or niobium powder are blended prior to deoxidation to form the alloy having an oxygen content less than 300 ppm. The alloys of the present invention still further include alloys of tantalum and/or niobium and an alloying element wherein the oxygen addition associated with the alloying element does not raise the oxygen content of the alloy above 300 ppm.

As described above, in the process for producing formed powder metal products of tantalum, niobium and their alloys, a tantalum, niobium, or alloy of tantalum or niobium, powder is, if needed, deoxidized, to an oxygen content of less than 300 ppm, without exposing the powder to a temperature greater than $0.7 T_H$, and then the powder is consolidated, without exposing the powder to a

temperature greater than $0.7 T_H$, to form a tantalum, niobium, or alloy product, having an oxygen content below 300 ppm, preferably between about 100 and 300 ppm.

According to the present invention, a formed
5 tantalum, niobium or alloy product, having an oxygen content below 300 ppm, may be produced from powder, having an oxygen content below 300 ppm, by any known powder metallurgy technique, utilized for tantalum, niobium and their alloys, provided that the metal is not
10 exposed to a temperature greater than $0.7 T_H$. Exemplary of these powder metallurgy techniques used for forming the metal products are the following, in which the steps are listed in order of performance. Any of the techniques may be utilized in the present invention,
15 provided that any sintering, heating, or other handling, of the metal does not expose the metal to a temperature greater than $0.7 T_H$:

1. Cold Isostatic Pressing, Sintering, Encapsulating, Hot Isostatic Pressing and Thermo-Mechanical Processing;
- 20 2. Cold Isostatic Pressing, Sintering, Hot Isostatic Pressing and Thermo-Mechanical Processing;
3. Cold Isostatic Pressing, Encapsulating, Hot Isostatic Pressing and Thermo-Mechanical Processing;

4. Cold Isostatic Pressing, Encapsulating and Hot Isostatic Pressing;
5. Encapsulating and Hot Isostatic Pressing;
6. Cold Isostatic Pressing, Sintering, Encapsulating,
- 5 Extruding and Thermo-Mechanical Processing;
7. Cold Isostatic Pressing, Sintering, Extruding and Thermo-Mechanical Processing;
8. Cold Isostatic Pressing, Sintering and Extruding;
9. Cold Isostatic Pressing, Encapsulating, Extruding
- 10 and Thermo-Mechanical Processing;
10. Cold Isostatic Pressing, Encapsulating and Extruding;
11. Encapsulating and Extruding;
12. Mechanical Pressing, Sintering and Extruding;
- 15 13. Cold Isostatic Pressing, Sintering, Encapsulating, Forging and Thermo-Mechanical Processing.
14. Cold Isostatic Pressing, Encapsulating, Forging and Thermo-Mechanical Processing;
15. Cold Isostatic Pressing, Encapsulating and Forging;
- 20 16. Cold Isostatic Pressing, Sintering and Forging;
17. Cold Isostatic Pressing, Sintering and Rolling;
18. Encapsulating and Forging;
19. Encapsulating and Rolling;
20. Cold Isostatic Pressing, Sintering and Thermo-
- 25 Mechanical Processing;

21. Spray Depositing;

22. Mechanical Pressing and Sintering; and

23. Mechanical Pressing, Sintering, Repressing and
Resintering.

5 Other combinations of consolidating, heating and
deforming may also be utilized.

The effectiveness and advantages of the products and
processes of the present invention will be further
illustrated by the following examples which are intended
10 to be illustrative in nature and are not to be construed
as limiting the scope of the invention.

EXAMPLES

The following analytical test procedures were
utilized to determine the properties of the powders and
15 formed products of the present invention:

Carbon Content:

Carbon content of the tantalum, niobium or alloy
powder was determined by a gas method, using a Leco 1R-12
Carbon Determinator, Leco #528-035 Crucibles,
20 Leco #501-263 Copper Metal Accelerator, and Leco #501-507
Carbon Standards (0.0066 + 0.0004% C), manufactured by
LECO Corporation, 3000 Lakeview Avenue, St. Joseph,
MI. 49805. The crucibles were placed in a muffle furnace
and ignited at 1000 degrees C for 1 hour and then allowed
25 to cool and stored in a clean dessicator. A 1.0 gram

sample of tantalum, niobium, or alloy powder was then transferred to a crucible. The tantalum, niobium, or alloy powder in the crucible was then covered with approximately 1 gram of copper metal accelerator.

5 Several crucibles containing only one scoop of copper metal accelerator, and several crucibles containing 1 gram of carbon standard and 1 gram of copper metal accelerator were also prepared, for instrument calibration, as blank samples and standard samples
10 respectively. To calibrate the Carbon Determinator successive blanks were analyzed and the carbon determinator Digital Voltmeter (DVM) reading was adjusted to show 0.000000% carbon. Next successive standard samples were analyzed and the carbon determinator DVM
15 reading was adjusted to show $0.0066 \pm 0.0004\%$ carbon. After calibration the crucible containing the tantalum, niobium or alloy powder, covered with copper metal accelerator was analyzed. The carbon determinator DVM reading for the tantalum, niobium or alloy sample
20 equalled the carbon content in parts per million.

Nitrogen and Oxygen Content:

The Nitrogen and Oxygen content of the tantalum, niobium or alloy powder were determined using a Leco TC-30 Oxygen Nitrogen Analyzer, Leco #760-414
25 Graphite Crucibles, manufactured and sold by

Leco Corporation, 3000 Lakeview Avenue, St. Joseph,
MI 49805 and 5.08cm (2 inches) wide by 0.635mm
(0.025 inch) thick nickel foil. The nickel foil was, cut
into 2.54cm (1 inch) by 2.54cm (1 inch) squares, cleaned
5 and formed into capsules. 0.2 grams of a sample were
transferred to each capsule and the capsule was closed and
crimped into the smallest possible volume. The Leco TC-30
Oxygen Nitrogen Analyzer, was first calibrated using blank
and tantalum standards of known oxygen and nitrogen
10 content, in a manner similar to the manner described above
for calibrating the carbon determinator, and then the
samples were run through the analyzer to generate ppm
oxygen and ppm nitrogen.

The following properties were determined in
15 accordance with the ASTM Test method shown in the
following chart:

	<u>Property</u>	<u>ASTM Test Method</u>
	Particle Size	B-214
	Pressed Density	B-212
20	Grain Size	E-112
	Transverse Rupture Strength	B-528
	Powder Flow Rate	B-213
	B.E.T. Surface Area	C-699
	Yield Strength	E-8
25	Tensile Strength	E-8
	% Elongation	E-8

Density of Formed Product:

The density of the formed product was calculated by measuring the weight and the dimensions, height, width etc. of the product. From the dimensions, the volume of the product was calculated in cubic centimetres. Density was then calculated by dividing the weight of the product by the volume of the product.

Percentage (%) of Theoretical Density:

The percentage of theoretical density of the product was calculated by dividing the density of the product by the theoretical density of the metal, for example 16.6 grams/cubic centimetre for Tantalum.

EXAMPLE 1

Example 1 illustrates the production of a tantalum powder having an oxygen content less than 300 ppm. A starting tantalum powder having an oxygen content of about 600 ppm, a carbon content of about 40 ppm, and a nitrogen content of less than 10 ppm, was blended with an amount of about 1% by weight magnesium. The resulting blend was heated at 850 degrees C (0.34 T) for 2 hours.

The magnesium, not reacted with the oxygen, was then removed by further heating the blend to 1000 degrees C (0.38 T) at a pressure of 1.333×10^{-1} Pa (0.001 Torr.)

Any remaining magnesium was removed by immersing the powder in nitric acid at room temperature. The powder was then washed in water and air dried. The resulting tantalum powder had an oxygen content of 185 ppm, a carbon content of 45 ppm, and a nitrogen content of 45 ppm. The resulting tantalum powder also had an apparent density of 4.12 gm/cc and a flow rate of 26 seconds for 50 grams. The particle size distribution was as shown below:

10	<u>Particle Size</u>	<u>wt. %</u>
	40/60	0.1%
	60/100	56%
	100/200	37.8%
	200/325	2.4%
15	325	3.7%

EXAMPLE 2

Example 2 illustrates a formed product of tantalum, having an oxygen content of about 205 ppm, produced by mechanical pressing and sintering.

20 A deoxidized tantalum powder having a carbon content of about 60 ppm, an oxygen content of about 135 ppm, and a nitrogen content of about 10 ppm, prepared by a procedure similar to the procedure of Example 1, was utilized as the starting powder. This tantalum powder
25 was placed in a die and pressed, using uniaxial pressure, into a 10.16cm (4 inch) diameter tablet with a pressed

density of about 80% of the theoretical density. This
tablet was then sintered at 1500 degrees C (0.54 T)
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for 2 hours in a vacuum evacuated to less than about
 1.333×10^{-2} Pa (.001 Torr). The final sintered tablet
5 had a carbon content of about 60 ppm, an oxygen content
of about 205 ppm and nitrogen content of about 10 ppm.

EXAMPLE 3

The following tests were conducted to show that the
tantalum, niobium or alloy powder, of the present
10 invention, is compressible, and to show the strength of
the powder of the present invention.

A deoxidized tantalum powder having a carbon content
of about 60 ppm, an oxygen content of about 135 ppm, and
a nitrogen content of about 10 ppm, prepared by a
15 procedure similar to the procedure of Example 1, was
utilized as the starting powder. The starting powder was
placed in a die and pressed at various pressures, into
tablets, 2.54cm (1 inch) in diameter, and about 1.27cm
(1/2 inch) in height. The density of the tablets as a
20 function of the Pressing pressures was as follows:

	Pressure		Density
	<u>Kgf/cm² x 10</u>	<u>(lbs/sq.in)</u>	<u>(% of theoretical)</u>
5	246.07	(35,000)	75.5
	281.23	(40,000)	78
	316.38	(45,000)	80
	351.53	(50,000)	82.1
	386.69	(55,000)	83.6
10	421.84	(60,000)	85.1
	456.99	(65,000)	86.4
	492.15	(70,000)	87.5
	562.46	(80,000)	89.7
	703.07	(100,000)	92.6

These results show that the powders of the present invention are compressible.

15 To show the strength of the powder of the present invention after mechanical pressing, a deoxidized tantalum powder having a carbon content of about 60 ppm, an oxygen content of about 135 ppm, and a nitrogen content of about 10 ppm, prepared by a procedure similar
20 to the procedure of Example 1, was placed in a die and pressed, at various pressures, into bars about 1.27 cm (1/2 inch) by about 1.27 cm (1/2 inch), by about 5.08 cm (2 inches). The transverse rupture strength of these bars was as follows:

	Pressure		Transverse Rupture Strength	
	<u>Kgf/cm² x 10</u>	<u>(lbs/sq.in)</u>	<u>Kgf/cm² x 10</u>	<u>(lbs/sq.in)</u>
30	140.61	(20,000)	7.73	(1100)
	210.92	(30,000)	13.36	(1940)
	260.14	(37,000)	19.12	(2720)
	421.84	(60,000)	54.12	(7700)

Generally a minimum strength of 140.6 Kgf/cm² (2000 lbs./sq.in.) is desired for normal handling of pressed compacts. The data from the compressibility test together with the rupture strength test indicates that this strength level can be obtained with the powder of the present invention formed at a pressure somewhat in excess of 30,000 psi, where the pressed compact has a density of about 75% of the theoretical.

EXAMPLE 4

Example 4 illustrates the production of a formed tantalum product having an oxygen content of about 130 ppm without exposing the metal to a temperature greater than 0.7 T_H, by cold isostatic pressing (CIP), followed by hot isostatic pressing (HIP) and finally followed by theremo-mechanical processing (TMP).

A deoxidized tantalum powder having a carbon content of about 10 ppm, an oxygen content of about 155 ppm, and a nitrogen content of about 15 ppm, prepared by a procedure similar to the procedure of Example 1, was utilized as the starting powder. This powder was cold isostatically pressed at 421.84 Kgf/CM² x 10 (60,000 lbs./sq.in.) and room temperature, into a preform of about 12.7cm (5.0 inches) by about 26.16cm (10.3 inches) by about 4.06cm (1.6 inches) with a weight of

about 22.66Kg (50 pounds). This preform was hermetically encapsulated then hot isostatically pressed at 295.29 Kg/cm² x 10 (42,000 lbs/sq.in), and 1300 degrees C (0.48 T_H) for 4 hours into a preform of about 12.07cm

5 (4.75 inches) by about 25.91cms (10.2 inches) by about 3.68cm (1.45 inches). The hot isostatically pressed preform had a carbon content of about 45 ppm, an oxygen content of about 130 ppm and a nitrogen content of less than about 10 ppm.

10 The hot isostatically pressed preform was then annealed at 1300 degrees C (0.48 T_H) for 2 hours in a vacuum evacuated to less than about 1.333×10^{-1} Pa (.001 Torr.) and then the encapsulation was removed. The resultant preform was rolled to a thickness (t) of about 15 1.02cm (0.4 inch). Then the rolled preform was annealed at 1300 degrees C (0.48 T_H) for 2 hours in a vacuum

evacuated to less than about 0.001 Torr. Next the preform was rerolled to a thickness (t) of about 2.03mm (0.08 inch). Then the rerolled preform was annealed at 20 1300 degrees C (0.48 T_H) for 2 hours in a vacuum

evacuated to less than about 0.001 Torr. Next the preform was rolled to a thickness (t) of about 0.38mm (0.015 inch). Then the three times rolled preform was

annealed at 1300 degrees C ($0.48T_H$) for 2 hours in a vacuum evacuated to less than about 1.333×10^{-1} Pa (0.001 Torr). Samples of the preform at various thickness were taken during process herein described.

5 The mechanical properties of the preform at the various thicknesses, in annealed condition, were as follows:

	<u>Condition</u>	<u>Yield Strength</u>		<u>Tensile Strength</u>		<u>Elong</u>	<u>Grain</u>
		<u>Kg/cm²</u> <u>x 10</u>	<u>(lbs./</u> <u>sq.in.)</u>	<u>Kg/cm²</u> <u>x 10</u>	<u>(lbs./</u> <u>sq.in.)</u>	<u>%</u>	<u>Size</u>
10	As HIPed	244.67	34,800	370.52	52,700	48	7
	t= 6.35mm (0.25 in.)	276.3	39,300	340.28	48,400	47	—
	t= 2.03mm (0.08 in.)	299.51	42,600	360.67	51,300	41	—
	t= 0.76mm (0.03 in.)	307.24	43,700	379.66	54,000	40	—
15	t= 0.38mm (0.015in.)	281.23	40,800	359.27	51,100	40	8

These properties are comparable to properties of tantalum sheet produced by sintering at a temperature greater than about $0.7 T_H$, which indicates that the powders and formed

products of the present invention are suitable for use in the same applications as products produced by sintering at a temperature greater than about $0.7 T_H$.

EXAMPLE 5

Example 5 illustrates the production of a formed tantalum product having an oxygen content of about 140 ppm, a carbon content of 30 ppm, and a nitrogen content of 15 ppm, without exposing the metal to a temperature greater than $0.7 T_H$ by cold isostatic pressing, sintering and then thermo-mechanical processing.

A deoxidized tantalum powder having a carbon content of about 10 ppm, an oxygen content of about 155 ppm, and a nitrogen content of about 15 ppm, prepared by a procedure similar to the procedure of Example 1, was utilized as the starting powder. This powder was Cold Isostatically pressed at $421.84 \text{ Kg/Cm}^2 \times 10$ (60,000 lbs./sq.in) into a bar shaped preform of about 1.6cm (0.63 inch) by about 6.35cm (2.5 inches) by about 63.5cm (25 inches) weighing about 11.34 Kg (25 pounds).

This preform was sintered at 1500 degrees C (0.53 T_H)

for 2 hours in a vacuum evacuated to less than about
1.333 x 10⁻¹ Pa (0.001 Torr), to yield a preform having a
density of about 95% of the theoretical density. The

5 preform was then rolled to a thickness (t) of about
0.51cm (0.2 inch) and a width of about 15.24cm (6 inches)
and a length of about 76.2cm (30 inches). Then the
rolled preform was annealed at 1300 degrees C (0.48 T_H)

for 2 hours in a vacuum evacuated to less than about
10 1.333 x 10⁻¹ Pa (0.001 Torr). The formed sheet had a
carbon content of 30 ppm, an oxygen content of 140 ppm,
and a nitrogen content of 15 ppm. The density of the
sheet was 100% of the theoretical density and the grain
size was 8.5. The longitudinal axis of the sheet had a
15 yield strength of 384.58 Kg/Cm² x 10 (54,700 lbs./sq.in),
a tensile strength of 281.23 Kg/Cm² x 10
(40,000 lbs./sq.in) and 45% elongation. The transverse
axis of the sheet had a yield strength of
380.36 Kg/Cm² x 10 (54,100 lbs./sq.in.), a tensile
20 strength of 257.32 Kg/Cm² x 10 (36,600 lbs./sq.in.) and
46% elongation. These results indicate that the sheet is
suitable for use in the same applications as sheets
produced by exposing tantalum to a temperature greater
than about 0.7 T_H.

EXAMPLE 6

Example 6 illustrates the production of a formed tantalum product having an oxygen content of about 205 ppm, a carbon content of 60 ppm, and a nitrogen content of 10 ppm, prepared without exposing the metal to temperature greater than 0.7 T_H by mechanical pressing, sintering, repressing and resintering.

A deoxidized tantalum powder having a carbon content of about 60 ppm, an oxygen content of about 135 ppm, and a nitrogen content of about 10 ppm, prepared by a procedure similar to the procedure of Example 1, was utilized as the starting powder. This tantalum powder was placed in a die and mechanically pressed, using uniaxial pressure, into a tablet, 7.62mm (0.3 inch) diameter by 0.36mm (0.14 inch) high. This tablet was then sintered at 1450 degrees C (0.53 T_H) for 2 hours in a vacuum evacuated to less than about 1.333×10^{-1} Pa (0.001 Torr). The final sintered tablet had a carbon content of about 60 ppm, an oxygen content of about 205 ppm and a nitrogen content of about 10 ppm.

The sintered tablet was then repressed into a preform. The preform was then resintered at 1450 degrees C (0.53 T_H) for 2 hours in a vacuum evacuated to less

than about 1.333×10^{-1} Pa (0.001 Torr). The resulting resintered preform was suitable for extruding to produce a formed tantalum product.

EXAMPLE 7

5 Example 7 illustrates the production of a formed tantalum product having an oxygen content of about 165 ppm, a carbon content of 90 ppm, and a nitrogen content of 10 ppm, prepared without exposing the metal to a temperature greater than 0.7 T_H by cold isostatic pressing, encapsulating and then extruding:

A deoxidized tantalum powder having a carbon content of about 80 ppm, an oxygen content of about 155 ppm, and a nitrogen content of less than about 10 ppm, prepared by a procedure similar to the procedure of Example 1, was
15 utilized as the starting powder. This tantalum powder was Cold Isostatically pressed at $421.84 \text{ Kg/cm}^2 \times 10$ (60,000 lbs./sq.in.) into a rod shaped preform of about 50.8cm (2 inches) in diameter by about 12.7cm (5 inches) long. The rod shaped preform was then hermetically
20 encapsulated in a steel container and extruded at 1150 degrees C (0.43 T_H) through a 1.59cm (5/8 inch) diameter die. The Encapsulating steel container was then removed and the preform was annealed at 1300 degrees C (0.48 T_H)

for 2 hours in a vacuum evacuated to less than about
1.333 x 10⁻¹ Pa (0.001 Torr). The annealed preform had a
carbon content of about 90 ppm, an oxygen content of
about 165 ppm, a nitrogen content of less than about
5 10 ppm, a yield strength of 292.48 Kg/Cm² x 10
(41,600 lbs./sq.in.), a tensile strength of
423.95 Kg/Cm² x 10 (60,300 lbs./sq.in.) and an elongation
of 52%. The annealed preform had a grain size of 12.5
microns.

10 The properties of the annealed preform indicate that
the annealed preform is suitable for subsequent thermo-
mechanical processing.

EXAMPLE 8

Example 8 illustrates the production of a formed
15 tantalum product having oxygen content of about 155 ppm,
prepared without exposing the metal to a temperature
greater than 0.7 T_H, by spray deposition.

A deoxidized tantalum powder having a carbon content
of about 80 ppm, an oxygen content of about 155 ppm, and
20 a nitrogen content of less than about 10 ppm, prepared by
a procedure similar to the procedure of Example 1, was
utilized as the starting powder. The powder was spray
deposited up to a thickness of 0.25mm (0.01 inch) on an

alloy substrate formed from Hastelloy Alloy X (Hastelloy is a trademark for alloys produced and sold by Haynes Corporation, Park Avenue, Kokomo, Indiana). No problems were encountered, indicating that the particle size, flow properties and oxygen content of the powder of the present invention are suitable for consolidation by spray deposition.

EXAMPLE 9

Example 9 illustrates the production of a niobium powder having an oxygen content of 175 ppm. The starting niobium powder having an oxygen content of about 660 ppm, a carbon content of about 25 ppm, and a nitrogen content of about 70 ppm, was blended with an amount of about 1.5% by weight magnesium. The resulting blend was heated at

850 degrees C (0.34 T_H) for 2 hours in an Argon

atmosphere. The magnesium, not reacted with the oxygen, was then removed by further heating the blend to

850 degrees C (0.34 T_H) at a pressure of 1.333×10^{-1} Pa

(0.001 Torr). Any remaining magnesium was removed by

immersing the powder in nitric acid at room temperature.

The powder was then washed with water and air dried. The resulting niobium powder had an oxygen content of

175 ppm, a carbon content of 20 ppm, and a nitrogen

content of 55 ppm. The resulting niobium powder also had

an apparent density of 3.45 gm/cc and a flow rate of 22 seconds for 50 grams. The particle size distribution was as shown below:

	<u>Particle size</u>	<u>wt. %</u>
5	60/100	-
	100/200	74%
	200/325	23%
	325/500	2%
	-500	1%

10 Numerous variations and modifications may obviously be made without departing from the present invention. Accordingly, it should be clearly understood that the forms of the present invention herein described are illustrative only and are not intended to limit the scope
15 of the invention.

CLAIMS

1. A metal powder having an oxygen content of less than 300 parts per million wherein the metal is selected from the group consisting of tantalum, niobium, an alloy of tantalum and an alloy of niobium.
2. The metal powder of claim 1 wherein the metal is tantalum.
3. The metal powder of claim 1 wherein the metal is niobium.
4. The metal powder of claim 1 wherein the metal is an alloy of tantalum.
5. The metal powder of claim 1 wherein the metal is an alloy of niobium.
6. A process for producing a metal powder having an oxygen content of less than 300 parts per million, wherein the metal is selected from the group consisting of tantalum, niobium, an alloy of tantalum and an alloy of niobium, without exposing the metal to a temperature greater than $0.7 T_H$ of the metal.
7. The process of claim 6 wherein the metal is tantalum.
8. The process of claim 6 wherein the metal is niobium.
9. The process of claim 6 wherein the metal is an alloy of tantalum.

10. The process of claim 6 wherein the metal is an alloy of niobium.

11. A formed powder metallurgy metal product having an oxygen content less than 300 parts per million produced without exposing the metal to a temperature greater than 0.7 T_H of the metal, wherein the metal is selected from

the group consisting of tantalum, niobium, an alloy of tantalum, and an alloy of niobium.

12. The formed powder metallurgy metal product of claim 11 wherein the metal is tantalum.

13. The formed powder metallurgy metal product of claim 11 wherein the metal is niobium.

14. The formed powder metallurgy metal product of claim 11 wherein the metal is an alloy of tantalum.

15. The formed powder metallurgy metal product of claim 11 wherein the metal is an alloy of niobium.

16. A process for producing formed powder metallurgy metal products, having an oxygen content less than 300 parts per million, wherein the metal is selected from the group consisting of tantalum, niobium, an alloy of tantalum and an alloy of niobium, comprising:

forming a metal product from the metal powder having an oxygen content less than 300 parts per million, without

exposing the metal to a temperature greater than $0.7 T_H$ of the metal.

17. The process of claim 16 wherein the metal is tantalum.

18. The process of claim 16 wherein the metal is niobium.

19. The process of claim 16 wherein the metal is an alloy of tantalum.

20. The process of claim 16 wherein the metal is an alloy of niobium.

21. A process for reducing the oxygen content of a metal powder wherein the metal is selected from the group consisting of tantalum, niobium, an alloy of tantalum, and an alloy of niobium, comprising: heating the metal powder to a temperature not greater than $0.7 T_H$ of the metal, in

the presence of a metal having a higher affinity for oxygen than the metal powder, for a period of time sufficient to reduce the oxygen content of the metal powder to less than 300 parts per million.

22. The process of claim 21 wherein the metal powder is tantalum.

23. The process of claim 21 wherein the metal powder is niobium.

24. The process of claim 21 wherein the metal powder is an alloy of tantalum.

25. The process of claim 21 wherein the metal powder is an alloy of niobium.

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